



Energy & Environmental Research Center

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July 29, 2020

Ms. Karlene Fine
Executive Director
North Dakota Industrial Commission
State Capitol, 14th Floor
600 East Boulevard Avenue, Department 405
Bismarck, ND 58505-0840

Dear Ms. Fine:

Subject: Quarterly Project Status Report Entitled "Low-Pressure Electrolytic Ammonia Production"; Contract No. R-036-45; EERC Fund 22946

Attached is a copy of the subject project status report for the period of April 1 through June 30, 2020.

If you have any questions, please contact me by phone at (701) 777-2982 or by e-mail at taulich@undeerc.org.

Sincerely,

DocuSigned by:
A blue ink signature of Ted R. Aulich, written in a cursive style.

89B1B8E4D0E7430...
Ted R. Aulich
Principal Process Chemist
Fuels and Chemicals

TRA/rlo

Attachment

c/att: Andrea Holl Pfennig, North Dakota Industrial Commission



LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION

Quarterly Project Status Report

(for the period of April 1, 2020, through June 30, 2020)

Prepared for:

Karlene Fine

North Dakota Industrial Commission
State Capitol, 14th Floor
600 East Boulevard Avenue, Department 405
Bismarck, ND 58505-0840

Contract No. R-036-45

Prepared by:

Ted R. Aulich

Energy & Environmental Research Center
University of North Dakota
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July 2020

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ACKNOWLEDGMENT

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LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION

Quarterly Project Status Report

April 1 – June 30, 2020

EXECUTIVE SUMMARY

This quarterly report summarizes April–June 2020 progress made toward achieving milestones and objectives of the low-pressure electrolytic ammonia (LPEA) project under way at the University of North Dakota Energy & Environmental Research Center (EERC). Partners on the 3-year (June 2018 – July 2021) project include North Dakota State University (NDSU), Nel Hydrogen (formerly Proton OnSite), and North Dakota Industrial Commission. The project goal is to demonstrate an ammonia production energy reduction of at least 16% by replacing state-of-the-art (2018) high-pressure Haber–Bosch-based ammonia synthesis with the EERC-developed LPEA process. Achieving this energy reduction goal requires improving the proton conductivity, gas impermeability, and durability of the EERC–NDSU-developed polymer–inorganic composite (PIC) proton exchange membrane, a critical LPEA process component capable of high-rate proton transfer at 300°C. Key accomplishments of the April–June 2020 quarter include the following:

- A titanium membrane sample holder was procured for measurement of proton conductivity, with the objective of eliminating suspected measurement problems resulting from oxide buildup on membrane contact surfaces of the stainless steel holder normally used. Unlike test outputs achieved with the stainless steel holder, tests conducted with the titanium holder consistently yielded high-quality Nyquist plots, required for accurate conductivity measurement.
- A bismuth-on-carbon black catalyst was synthesized, characterized, and evaluated for application as cathode catalyst. In room-temperature chronoamperometric tests, the catalyst yielded an ammonia synthesis rate of $36.4 \mu\text{g}\cdot\text{h}^{-1}\cdot\text{mg}^{-1}$ at a faradaic efficiency of 7.9 %.
- PIC membrane-based MEA (membrane electrode assembly) fabrication method development work was initiated using the following key components:
 - Solution-cast 60- μm -thick PIC membrane comprising 75 wt% IPC (inorganic proton conductor) particles (sized via a sedimentation rate-based technique) and 25 wt% PBI (polybenzimidazole).
 - NbN cathode catalyst.
 - Pt anode catalyst.

The EERC holds an unwavering commitment to the health and well-being of its employees, partners and clients, and our global community. As such, precautionary measures have been implemented in response to COVID-19. Staff continue to carry out project-related activities remotely, and personnel supporting essential on-site laboratory and testing activities are proceeding under firm safety guidelines. Travel has been minimized, and protective measures are being undertaken for those who are required to travel. At this time, work conducted by EERC

employees is anticipated to progress with minimal disruption. Challenges posed by economic variability will be met with open discussion between the EERC and project partners to identify solutions. The EERC is monitoring developments across the nation and abroad to minimize risks, achieve project goals, and ensure the success of our partners and clients. In the event that any potential impacts to reporting, scope of work, schedule or cost are identified, they will be discussed and addressed in cooperation with the project partners.

LOW-PRESSURE ELECTROLYTIC AMMONIA PRODUCTION

Quarterly Project Status Report

January 1 – March 31, 2020

PROJECT GOALS/OBJECTIVES

The project goal is to demonstrate an ammonia production energy reduction of 16% by replacing state-of-the-art (2018) high-pressure Haber–Bosch (HB)-based ammonia synthesis with the Energy & Environmental Research Center (EERC)-developed low-pressure electrolytic ammonia (LPEA) process, as shown in Figure 1. To achieve the 16% production energy reduction target will require improving the LPEA process, which will require improving the polymer–inorganic composite (PIC) proton exchange membrane (PEM) on which the LPEA electrochemical cell is based. As a result, the proposed project is focused on improving the performance and durability of the PIC membrane, with the objective of producing a membrane that exhibits the following properties:

- Proton conductivity of $\geq 10^{-2}$ Siemens/centimeter (S/cm) and gas permeability of $< 2\%$ at a minimum temperature of 300°C .
- Ability to sustain 10^{-2} S/cm proton conductivity for at least 1000 hours (h).
- Mechanical strength (at 300°C) comparable to that of a commercial proton exchange-based electrolyzer membrane.

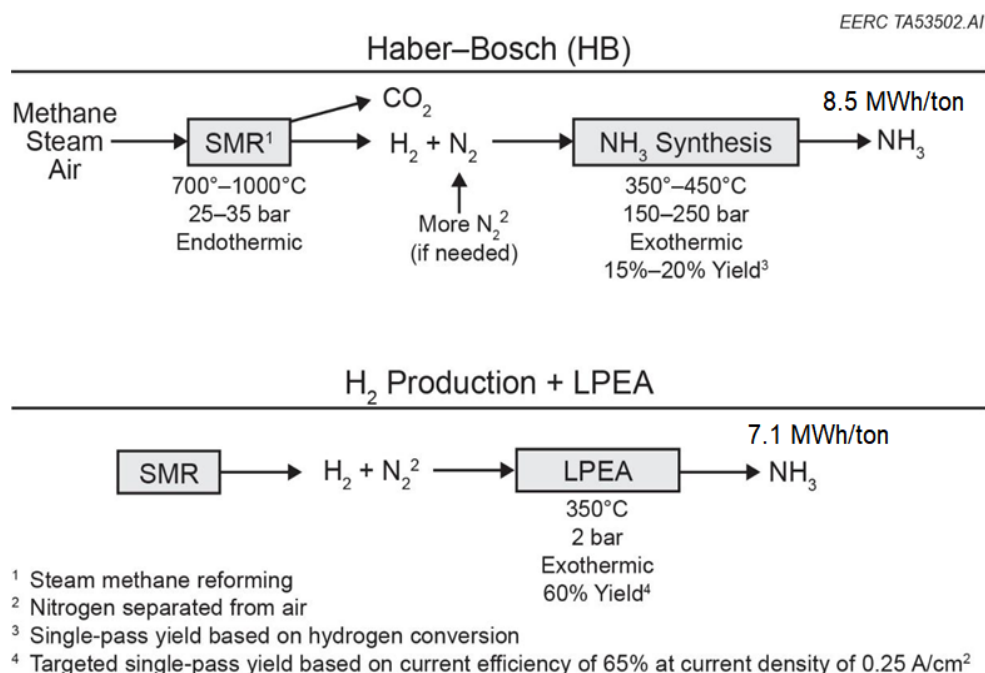


Figure 1. State-of-the-art (2018) HB versus LPEA-based NH₃ production.

- As measured in an MEA (membrane electrode assembly) at a minimum temperature of 300°C, current efficiency of $\geq 65\%$ for NH_3 formation at a current density of $\geq 0.25 \text{ amps/cm}^2$ (A/cm^2), NH_3 production energy efficiency of $\geq 65\%$, and $\leq 0.3\%$ performance degradation per 1000 h of operation.

BACKGROUND

In support of U.S. Department of Energy (DOE) Energy Efficiency and Renewable Energy (EERE) Advanced Manufacturing Office (AMO) goals to reduce life cycle energy consumption of manufactured goods and more cost-effectively use hydrogen in manufacturing processes, this project is focused on optimizing and demonstrating the improved efficiency (versus HB ammonia production) of the EERC-developed LPEA production process. Because it does not require the high pressure and high recycle rate (because of low single-pass ammonia yield) of the HB process, LPEA offers the potential for significant reduction in both energy consumption and cost. Partners on the proposed project are North Dakota State University (NDSU), Nel Hydrogen (Nel) (formerly Proton OnSite), the University of North Dakota Chemistry Department (UND Chemistry), and the North Dakota Industrial Commission (NDIC). The LPEA process is based on an innovative EERC-developed PIC high-temperature PEM. The process operates at ambient pressure and a temperature of 300°C and uses inputs of hydrogen, nitrogen, and electricity to make ammonia. The EERC demonstrated LPEA process viability in ammonia formation tests conducted using a 0.2-watt electrochemical cell built around an early-stage PIC membrane.

To meet the above-listed membrane performance and durability specifications, the project initially targeted fabrication—via a “co-electrospinning” technique—of a PIC membrane comprising “core-shell” inorganic proton conductor–polybenzimidazole (IPC–PBI) proton-conducting nanofibers contained within and aligned perpendicularly to the plane of a PBI matrix/membrane, as shown in Figure 2. Because each fiber core would comprise a chain of IPC particles in contiguous contact with one another throughout the chain length, each fiber would essentially function as a high-efficiency proton transport conduit running straight through the membrane. However, during Budget Period 1 (BP1) of the project, an alternative IPC was identified that offered significantly improved proton conductivity, stability, and durability—at 300°C—than the originally proposed IPC. Because this new IPC encompasses chemical and physical properties not readily amenable to co-electrospinning with PBI to yield core-shell nanofibers, new methods for IPC deployment in PBI matrix are being pursued. Primary focus is on film-casting (also referred to as solution-casting) a colloidal suspension of optimally sized IPC particles in a solution comprising PBI dissolved in dimethylacetamide (DMAc).

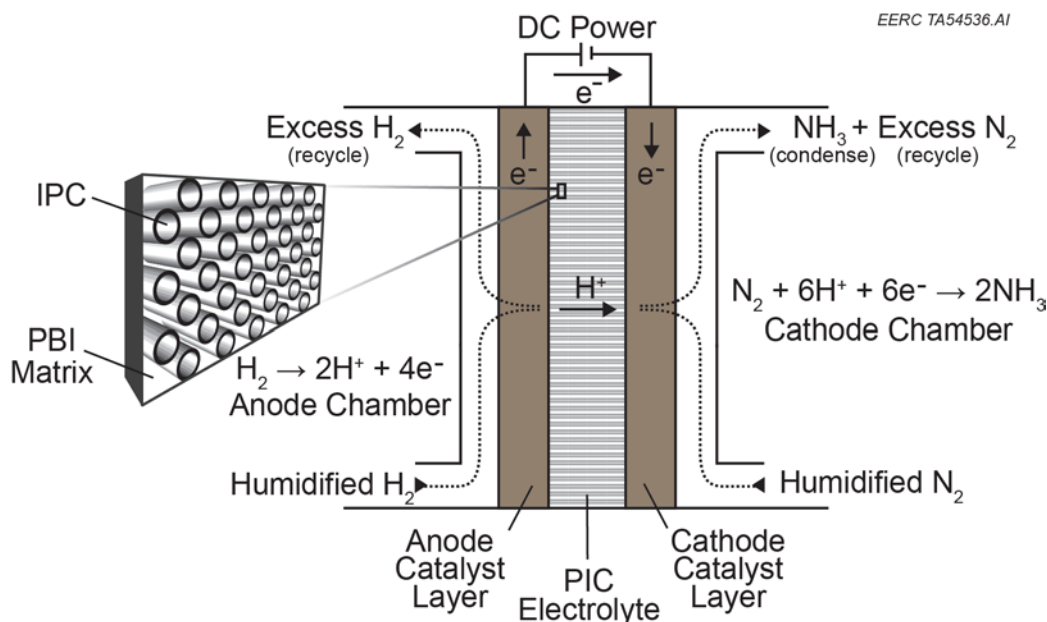


Figure 2. LPEA process.

Following fabrication of a PIC membrane that meets performance and durability specifications, the membrane—along with selected anode and cathode catalysts—will be used to construct experimental MEAs. MEAs will be incorporated into LPEA unit cells that will be evaluated based on NH_3 formation efficiency and durability, with the objective of identifying an optimal MEA configuration. The optimal MEA configuration will be used as the basis for building a stack of several LPEA unit cells that will compose an LPEA system capable of producing at least 100 grams/day (g/d) of NH_3 . The 100-g/d LPEA system will undergo optimization and then be used to demonstrate NH_3 synthesis (from H_2) at the LPEA target production energy input requirement of 0.8 megawatt hours (MWh/ton), which would translate to a total (H_2 production plus NH_3 synthesis) LPEA-based NH_3 production energy input requirement of 7.1 MWh/ton, the project-targeted goal. LPEA system operation and performance data will be used to perform a techno-economic evaluation of the LPEA-based NH_3 production process.

ACCOMPLISHMENTS

- A titanium membrane sample holder was procured for measurement of proton conductivity, with the objective of eliminating suspected measurement problems resulting from oxide buildup on membrane contact surfaces of the stainless steel holder normally used. Unlike test outputs achieved with the stainless steel holder, tests conducted with the titanium holder consistently yielded high-quality Nyquist plots, required for accurate conductivity measurement.

- A bismuth-on-carbon black catalyst was synthesized, characterized, and evaluated for application as cathode catalyst. In room-temperature chronoamperometric tests, the catalyst yielded an ammonia synthesis rate of $36.4 \mu\text{g}\cdot\text{h}^{-1}\cdot\text{mg}^{-1}$ at a faradaic efficiency of 7.9%.
- PIC membrane-based MEA fabrication method development work was initiated using the following key components:
 - Solution-cast 60- μm -thick PIC membrane comprising 75 wt% IPC particles (sized via a sedimentation rate-based technique) and 25 wt% PBI.
 - NbN cathode catalyst.
 - Pt anode catalyst.

PROGRESS AND STATUS

Task 1 – Project Management

Table 1 summarizes project task status. Tasks 2 and 4 are not included, since remaining activities from these tasks were incorporated into Tasks 3 and 5, respectively, at the conclusion of BP1. As shown, all tasks are behind schedule. These progress delays resulted from 1) a tentative BP2 start while awaiting official approval of BP2 funding and 2) a directive issued March 15 by UND President Dr. Joshua Wynne (in response to coronavirus spread concerns) instructing all nonessential EERC employees to work remotely till further notice, which restricted project laboratory activities. Similar restrictions were implemented at roughly the same time by project partners NDSU and Nel. Recently, labs at all project locations were reopened—with social distancing-based staffing capacity limits. Because the project is 12 months from planned end date, the EERC and partners will attempt to bring the project back on schedule and defer any decision on an extension request until at least the end of the current quarter (September 2020). Two important Task 1 accomplishments include:

- Publication of “A Comparative Experimental Study of the Hygroscopic and Mechanical Behaviors of Electrospun Nanofiber Membranes and Solution-cast Films of Polybenzimidazole” (primarily authored by LPEA project team members Xiangfa Wu and Oksana Zholobko of NDSU) in the current issue of *Journal of Applied Polymer Science*. Of significance is that a photo accompanying the article was selected as the issue cover photo.
- Submittal to the U.S. Patent Office of a utility patent application (Serial Number 16/922,160) for the project-developed 300°C-capable PEM comprising an IPC composited with PBI. The patent application describes membrane synthesis, properties, performance, and applications.

Table 1. Task Schedule – BP2

Task No.	Task Description	Task Completion Date			Task Progress Notes
		Original Planned	Revised Planned	% Complete	
1	Project Management	14 June 2021		58	
3	Optimize IPC and PIC membrane performance and durability	14 Dec. 2020		65	Behind schedule
5	Screen cathode catalysts, fabricate MEAs, deploy MEAs in unit cell for LPEA process optimization	14 Dec. 2020		40	5 catalysts screened; MEA fabrication and unit cell work behind schedule
6	Design, fabricate, and optimize 100-g/d LPEA system; acquire data for techno-economic analysis	14 March 2021			Not started
7	Conduct techno-economic analysis	14 June 2021			Not started

Task 3 – Optimize IPC and PIC Membrane Performance and Durability

- A thin disk comprising 94 weight% (wt%) IPC2 was produced by a newly conceptualized hot-pressing technique using PBI powder (rather than PBI dissolved in dimethylacetamide) as a binder. The objective was to eliminate voids/cavities and increase packing density of IPC particles as compared to solution/film-cast membranes. In accordance with vendor (PBI Performance Products) recommendations, the disk was pressed at 480°C (PBI glass transition temp is 427°C) and 10,000 psi. Calculated volume percentages for the disk were 86% IPC2 and 14% PBI, assuming no void space remaining in the disk. The hot-pressed disk was hard and appeared to have very low porosity but has not yet been tested for proton conductivity. Unfortunately, the pressing die metallurgy was inadequate to handle the pressing conditions and the die sustained significant damage, rendering it inoperable. A pressing die set made of 316 stainless steel was ordered and recently received. The die will be utilized to produce additional PIC electrolyte thin disks.
- A literature survey was performed to identify ceramic binders with potential for replacing PBI as matrix for compositing IPC2 particles in a thin-disk proton exchange electrolyte. A possible binder with a glass transition temperature near that of PBI was identified. Unlike PBI, which exhibits essentially zero proton conductivity, the identified inorganic binder—based on its composition and structure—appears likely to offer significant proton conductivity. Chemical supplies were purchased and initial tests of production methods for making the ceramic binder were performed during this reporting period.

A titanium membrane sample holder was procured for measurement of proton conductivity, with the objective of eliminating suspected measurement problems resulting from oxide buildup on membrane contact surfaces of the stainless steel holder normally used. Unlike test outputs achieved with the stainless steel holder, tests conducted with the titanium holder consistently yielded high-quality Nyquist plots. These results indicate the necessity of conducting proton conductivity testing with the titanium holder to ensure maximum accuracy of results. Table 2 shows results of proton conductivity tests conducted with two different film/solution-cast membrane samples and one pressed disk prepared at room temperature using a “dough” comprising IPC2 particles mixed with a viscous solution of PBI in DMAc.

Table 2. Proton Conductivity of PIC Membranes/Disks at 300°C

Membrane Type	No. of Samples Tested	IPC2/PBI wt% Ratio	Thickness (microns)	Proton Conductivity (S/cm)
Solution	3	75/25	72	0.2×10^{-2}
Cast	1	85/15	92	0.3×10^{-2}
Disk	2	90/10	950	0.7×10^{-2}

Task 5 – Catalyst Screening and MEA/Unit Cell Development and Optimization

A literature-identified bismuth-based catalyst was prepared, characterized, and evaluated for nitrogen electroreduction activity. The “Bi-on-carbon black” catalyst was reported to exhibit a nitrogen reduction faradaic efficiency of 67% and ammonia yield of $200 \text{ mmol} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$ ($0.052 \text{ mmol} \cdot \text{cm}^{-2} \cdot \text{h}^{-1}$) in aqueous electrolyte under ambient conditions (Hao, 2019). To prepare the catalyst, 10 mg carbon black was fully dispersed in 10 mL of anhydrous ethylene glycol and sonicated for 2 hours, after which 5 mL of a solution comprising 2.8 mM $\text{Bi}(\text{NH}_3)_3 \cdot 5\text{H}_2\text{O}$ in ethylene glycol was added. The mixture was vigorously stirred for 16 hours, sonicated for another 15 minutes, and transferred to a 25-mL Teflon-lined stainless steel autoclave. After solvothermal reaction at 180°C for 16 hours, the final product was collected by centrifuge, washed with ethanol three times, and then dried. Energy-dispersive spectroscopy (EDS) was used to characterize the catalyst and confirm the presence of bismuth on carbon, as shown in Figure 3.

The Bi catalyst was screened for use as LPEA cathode catalyst. Screening tests were conducted with an Autolab potentiostat using a divided H-type electrochemical cell separated by ion exchange membrane (Nafion[®] 117). The Nafion membrane was pretreated in 5% H_2O_2 solution for 1 hour, then in $0.5 \text{ mol L}^{-1} \text{H}_2\text{SO}_4$ for 1 hour at 80°C, and then rinsed in ultrapure water several times. Catalyst inks were prepared by ultrasonically dispersing 10 mg of catalyst powder in a mixture composed of 450 μL isopropyl alcohol and 50 μL 10-weight% Nafion[®] solution. A catalyst loading of about $3 \text{ mg catalyst} \cdot \text{cm}^{-2}$ was deposited onto Toray carbon paper (3.5-cm^2 geometric area). Reference and counter electrodes were Ag/AgCl (saturated KCl) and platinum wire, respectively. During electrolysis, N_2 gas (99.99% purity) was continuously fed into the cathodic compartment. Figure 4 compares chronoamperometry curves obtained for the catalyst at four different constant potentials versus Ag/AgCl in N_2 -saturated $0.1 \text{ mol} \cdot \text{L}^{-1} \text{H}_2\text{SO}_4$ electrolyte, at room temperature. Table 3 compares catalyst performance at the four different potentials based on faradaic efficiency and ammonia production rate.

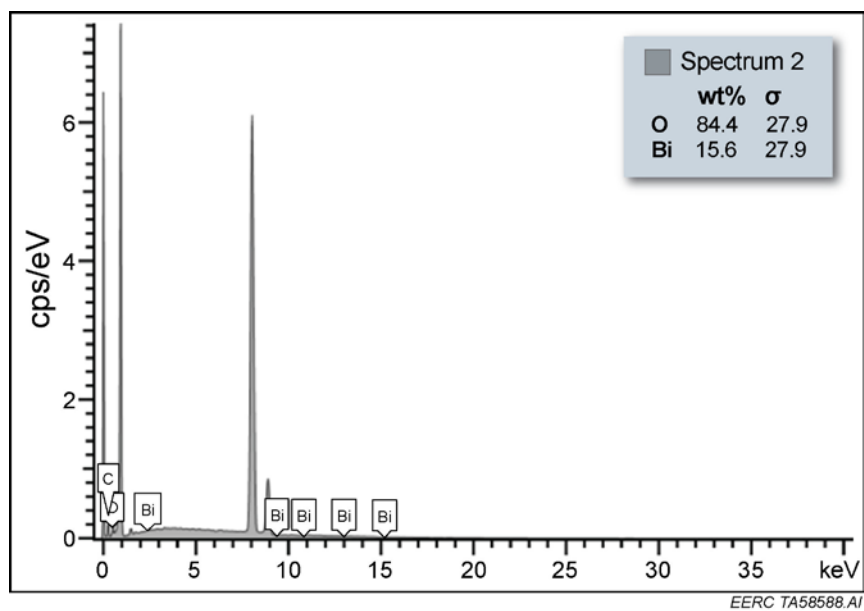


Figure 3. EDS analysis indicating presence and wt% of bismuth and oxygen. Other large peaks are for carbon (support) and copper (sample holder).

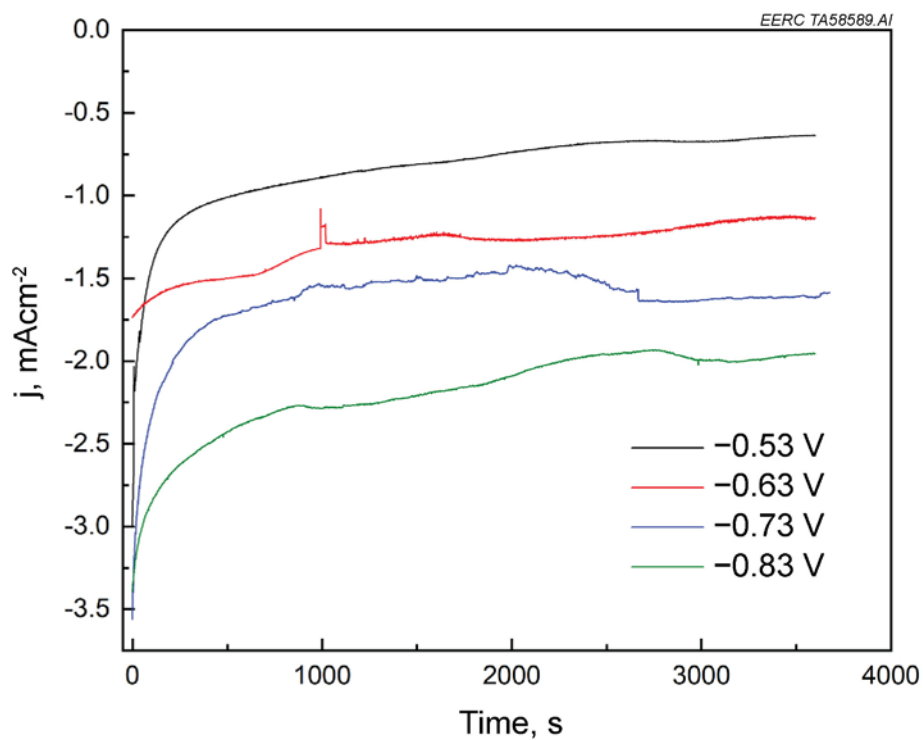


Figure 4. Chronoamperometry curves of thin-layer Bi/C catalyst recorded at -0.53 V , -0.63 V , -0.73 V and -0.83 V in N_2 -saturated $0.1\text{ mol}\cdot\text{L}^{-1}\text{ H}_2\text{SO}_4$ at room temperature.

Table 3. Nitrogen Reduction Performance of Bi-on-Carbon Black Catalyst

Applied Potential (V ¹ vs. RHE ²)	Absorbance Intensity	C _{NH4+} (μg/mL)	C _{NH3} (mol/L)	Coulombic Charge (C)	FE ³ (%)	Production Rate (μg h ⁻¹ mg _{cat} ⁻¹)	Production Rate (μmol h ⁻¹ mg _{cat} ⁻¹)
Mass loading of catalyst: 10 mg; Volume of catholyte: 0.015 L Electrolysis time: 1hour; Electrode area: 0.785 cm ² x2 = 1.57cm ² ; Potential (V vs. RHE)							
-0.53V	0.3299	0.7489	4.15E-05	2.71	6.65	19.3	0.0622
-0.63V	0.3244	0.7347	4.07E-05	3.34	5.29	18.7	0.0611
-0.73V	0.5964	1.4357	7.95E-05	4.37	7.91	36.37	0.1193
-0.83V	0.3592	0.8244	4.57E-05	9.42	2.10	21.06	0.0685

¹ Volts.² Reversible hydrogen electrode.³ Faradaic efficiency.

Table 3 shows that measured faradaic efficiencies and NH₃ production rates were significantly below reported values. Likely contributors to these results are:

- In the reported work, bismuth was deposited on carbon black as nanocrystals with an average size of 7.4 nm, while in our work, we were unable to confirm bismuth crystallinity and size because of scanning electron microscope technical and scheduling difficulties. According to the article, increasing bismuth particle size from 7.4 to >100 nm translated to faradaic efficiency and NH₃ production rate decreases of at least 40%.
- In the reported work, varying levels of K₂SO₄ were included in the electrolyte, whereas our work used only H₂SO₄ as electrolyte. As reported in the article, when K⁺ concentration in electrolyte was raised from 0.2 to 1.0 mol·L⁻¹, N₂ reduction current density increased from 0.14 to 0.50 mA·cm⁻², whereas H₂ evolution current density declined from 1.31 to 0.25 mA·cm⁻², thus increasing N₂ reduction faradaic efficiency from 9.8 to 67%.

PIC membrane-based MEA fabrication method development work was initiated using the following key components:

- Solution-cast 60-μm-thick PIC membrane comprising 75 wt% IPC (as “smaller particle-size fraction” recovered from sedimentation rate-based particle-size separation technique) and 25 wt% PBI.
- NbN cathode catalyst.
- Pt anode catalyst.

Task 6 – Design, Fabrication, and Operation of 100-g/d LPEA System

No activity this quarter.

Task 7 – Techno-Economic Analysis

No activity this quarter.

PLANS FOR NEXT QUARTER

Task 3 – Optimize IPC and PIC Membrane Performance and Durability

- Initial testing of the hot-pressing method using the stainless steel die set will focus on determining the porosity of pressed disks of IPC2 and the density of PBI at 480C. These tests will be done to determine the precise ratio of IPC2 to PBI that should be used to give a maximally dense proton conducting disk. If these measurements can be made accurately, then we believe that a disk could be prepared that will retain residual compressive stresses upon cooling. Much like in prestressed concrete, these residual stresses should significantly increase the strength of the hot-pressed disks, allowing us to make thinner disks that can stand up to the stresses occurring during normal handling of membranes during ammonia production. Once these measurements are made, hot pressed disks will be made with slightly varying ratios of PBI to IPC2 and their proton conductivities will be measured. In addition, we will begin to assemble the apparatus necessary to measure the flexural strengths of the disks.
- Additional testing will be done to develop ways to make the ceramic binder which has a sufficiently low glass transition temperature to allow for hot-pressing disks with IPC2 powder using our existing equipment. Proton conductivity of the binder will be measured. Tests similar to those being done with the PBI binder will be performed to create all-ceramic disks with residual compressive stresses that should be more resistant to degradation when making ammonia than the PBI-based membranes.

Task 5 – Catalyst Screening and MEA/Unit Cell Development and Optimization

- Using titanium membrane holder, proton conductivity and gas permeability testing will be conducted on membranes, hot-pressed disks made with PBI, and (possibly) disks prepared using inorganic IPC binder.
- MEA fabrication technique development will continue, with the objective of providing at least one PIC membrane-based MEA for proton conductivity testing and—importantly—ammonia synthesis testing at 300°C.

Task 6 – Design, Fabrication, and Operation of 100-g/d LPEA System

- Initiate design of 100-g/d system.

Task 7 – Techno-Economic Analysis

- Develop strategy/deployment scenario for economically competitive initial entry of LPEA into the commercial ammonia industry.

PRODUCTS

None.

IMPACTS

Impact on Technology Transfer and Commercialization Status

No commercialization impacts, progress, issues, or concerns to report during this quarter.

Dollar Amount of Award Budget Being Spent in Foreign Country(ies)

No spending of any project funds in any foreign countries has occurred or is planned.

CHANGES/PROBLEMS

The EERC is operational and open for business. Personnel that are not essential for on-site operations have transitioned to working from home. Essential project, laboratory, and field-based activities are proceeding with the incorporation of the Centers for Disease Control and Prevention, North Dakota State, and UND guidelines associated with COVID-19, and mitigation measures have been implemented.

In collaboration with project partners, the EERC is continually assessing potential impacts to project activities resulting from COVID-19 and/or the U.S. economic situation.

In the event that any potential impacts to reporting, scope of work, schedule, or cost are identified, they will be discussed and addressed in cooperation with the DOE Project Manager.

Scope Issues, Risks and Mitigation Strategies

None.

Actual or Anticipated Problems or Delays and Corrective Actions or Plans to Resolve Them

The project is behind schedule due to laboratory staffing restrictions imposed to minimize coronavirus spread. As a result of recent partial lifting of restrictions, the presence of two researchers in the EERC lab utilized for LPEA activities is now permitted—up from one, but well below typical pre-coronavirus (unrestricted) occupancies of six or more. Lab activities are

being regularly reviewed and prioritized to ensure maximum coordination between labs at EERC, NDSU, and Nel.

Changes That Have a Significant Impact on Expenditures

None.

RECIPIENT AND PRINCIPAL INVESTIGATOR DISCLOSURES

None.

CONFLICTS OF INTEREST WITHIN PROJECT TEAM

None.

PARTNERS AND FINANCIAL INFORMATION

This project is sponsored by NDIC, DOE, UND Chemistry, NDSU, and Proton. Table 4 shows the total budget of \$3,164,010 for this project and expenses through the reporting period.

Table 4. Project-to-Date Financial Report at June 30, 2020

Funding Source	Budget	Current Reporting Period Expenses	Cumulative Expenses as of 6/30/20	Remaining Balance
DOE	\$2,497,983	\$159,065	\$1,519,286	\$978,697
UND Chemistry – In Kind	\$69,027	\$18,036	\$61,280	\$7,747
NDIC	\$437,000	\$36,275	\$291,847	\$145,153
NDSU – In Kind	\$120,000	\$49,446	\$120,000	\$0
Proton – In Kind	\$40,000	\$7,897	\$13,507	\$26,493
Total	\$3,164,010	\$270,719	\$2,005,920	\$1,158,090

REFERENCE

Hao, Y.; Guo, Y.; Chen, L.; *et al.* Promoting Nitrogen Electroreduction to Ammonia with Bismuth Nanocrystals and Potassium Cations in Water. *Nature Catalysis* **2019**, 2, 448–456. <https://doi.org/10.1038/s41929-019-0241-7>.